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Effect of Crystal Anisotropy on the Mechanical Properties of WC Embedded in WC-Co Cemented Carbides

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Abstract

In this work the hardness, Young modulus and fracture toughness of micro-sized tungsten carbide particles (10-20 μm) embedded in a cobalt matrix (WC-Co cemented carbide) has been evaluated by means of nanoindentation. Hardness and Young modulus of these hard particles have been determined using a *Berkovich* tip indenter. Fracture toughness has been assessed by means of the indentation microfracture method using a cube corner indenter. The obtained hardness and fracture toughness values show a broad scatter due to the marked anisotropy of the tungsten carbide crystals. Results are analyzed by further identification of indented crystallographic planes by means of electron back-scattered diffraction. A correlation is established between hardness and fracture toughness on one side, and crystallographic orientation of the carbides on the other one.

1. Introduction

WC-Co cemented carbides, also referred as hardmetals, are materials often used as tools, structural components and wear parts with stringent requirements. They exhibit an exceptional combination of strength, toughness and wear resistance as a result of the extremely different properties of their two constitutive phases: hard and faceted WC grains embedded in a soft and ductile Co rich binder. Mechanical properties of hardmetals, especially hardness and toughness, strongly depend on the nature and size of each microstructural constituent. In this regard, although mixtures of different metallic binders (Co, Ni, Fe,...) and carbides (WC, TiC, TaC, etc.) effectively results in enhanced properties for particular service conditions, the optimal combination of mechanical parameters achieved by the plain WC-Co grades keeps them as forefront reference of the cemented carbides family [1]. The main role of WC in the microstructure is to provide hardness and wear resistance. On the other hand, the constrained plastic deformation and rupture of the tough Co binder phase ligaments, as well as the fracture of the carbide-binder interfaces mainly accounts for the global fracture toughness [2]. Additionally, the inter- and trans-crystalline fracture of WC should also be considered regarding energy absorption associated with unstable crack growth. Although WC rupture is believed to have a minor contribution, inter-crystalline fracture may be relevant for toughness accounting by effective development of crack deflection. The latter is a well known toughening mechanism that specially enhances toughness by inducing a change in the cracking path along large and faceted tough phases. Accordingly, it can be speculated that the effective toughness exhibited by carbides, as directly related to chemical nature or indirectly associated with relative crystal orientation, may affect the relative amount of crack deflection as the propagating cracks interact with the microstructure before final rupture. Thus, the evaluation of crystal anisotropy effects on toughness in WC crystals may be pointed out as another key micromechanical feature to understand the global mechanical response of WC-Co hardmetals.

WC presents a hexagonal crystal structure (type P-6m2), with three types of facets: two prismatic {10-10} and one basal (0001) (Figure 1) [3]. Previous works on WC single crystals show a marked anisotropy in hardness, being about 1450 HV for the prismatic planes and about 2500 HV for the basal ones [4]. Such pronounced anisotropy is expected to be found in other mechanical properties, as fracture toughness. However, to the best knowledge of the authors, experimental information on this parameter as a function of crystal orientation does not exist in the open literature. Accordingly, micromechanical characterization of WC crystals should also include such anisotropy and the obtained results must be analyzed in terms of crystal orientation. This is here approached by assessing the mechanical response of the WC phase as embedded in the hardmetal microstructure, where the coexistence of Co binder and WC yields, as a consequence of the processing route followed, a particular residual stress state that may affect the fracture properties of the WC.

Considering the micrometer-scale size of WC particles, nanoindentation emerges as suitable technique for their mechanical characterization in terms of hardness, elastic modulus and fracture toughness. Hardness and elastic modulus are routinely measured by nanoindentation [5]. On the other hand, fracture toughness may also be estimated by using this technique in hard phases as far as cracks are induced at the corners of the permanent imprint. In a recent study, this approach has been successfully implemented by the authors for characterizing primary carbides in tool steels [6]. It is the aim of this work to evaluate the effect of crystal anisotropy on the mechanical properties (hardness, elastic modulus and fracture toughness) of WC crystals, embedded in a hardmetal microstructure, by means of nanoindentation. Results are expected to shed light on the influence of the microconstituent properties on the microstructure-crack interaction and the global fracture resistance of hardmetals.

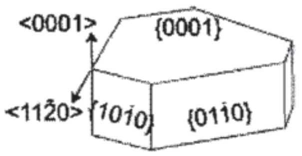


Figure 1. Morphology of WC grains in WC-Co alloys showing the (0001) basal and {10-10} prismatic facets.

2. Experimental procedure

2.1. Materials

The chemical composition and mechanical properties of the hardmetal used are shown in Table 1. Microstructure contains a quite large WC grain size (mean value about 20 microns), which allows the evaluation of hardness (H), elastic modulus (E) and fracture toughness (K_C) by nanoindentation.

Table 1. Chemical composition and hardness of the hardmetal studied.

Hardmetal	
WC (in wt %)	91.5
Co (in wt %)	8.5
Mean grain size (μm)	20.0
Hardness (HV)	1050

2.2. Mechanical characterization of WC crystals

Hardness (H) and elastic modulus (E) of WC were measured by means of nanoindentation using a Nanoindenter XP (MTS) system fitted with a *Berkovich* diamond tip. The tip was calibrated on a fused silica sample using the Oliver and Pharr method [5]. The indentations were carried out up to applied loads of 250 mN. A Poisson's ratio of 0.25 was assumed for WC crystals. Fracture toughness of WC was evaluated by means of the indentation microfracture (IM) method. Cracks of controlled size and shape can be induced in most hard materials by the application of sharp indenters. The cracks emanating from the corners of the indentation imprint are arrested when the residual stress driving force at the crack tip is in equilibrium with the K_C. Thus, applying equation (1), K_C can be readily measured in brittle materials by measuring the length of these indentation cracks.

$$K_c = \chi_v \left(\frac{E}{H}\right)^{2/5} \left(\frac{a}{l}\right)^{1/2} \frac{P}{c^{3/2}} \quad (1)$$

In equation (1) P is the indentation load, c is the crack length, a is the half-diagonal of the indentation impression , E is the Young's modulus, H is the hardness and χ_υ is a constant empirically determined that depends on the crack morphology and the indenter geometry. Moreover, the final morphology of the indentation cracks depends on the indentation load, the indenter tip geometry and the toughness of the material. Expression (1) was developed by Laugier for WC-Co materials, in which Palmqvist crack configuration (Figure 2) is generated by the application of a Vickers indenter [7]. For this configuration of crack morphology and indenter geometry, χ_υ was determined as 0.015. The *Berkovich* indenter is generally used in small-scale nanoindentation studies. Main reason behind it resides on the advantage that three-side pyramid edges are more easily constructed to meet at a single point than the inevitable line that occurs in the four-side Vickers pyramid. However, in relatively tough materials (K_C > 5-6 MPa·m$^{1/2}$) the *Berkovich* tip does not generate cracks at the imprint corners at low loads. In those cases, the cube corner tip, sharper than the *Berkovich* tip and then able of displacing more volume of material (more than three times) than the former, is preferred. The use of this indenter results in higher stresses below the contact area, allowing then fracture at lower loads. As a consequence, cube corner tips are usually applied to induce cracks in tougher materials and are therefore used in this work to indent WC single crystals. The value of χ_υ in equation (1) for a cube corner indenter has been calibrated following the methodology proposed by Casellas et al [6], giving a value of 0.057.

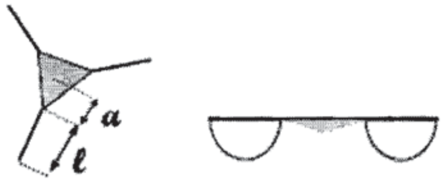
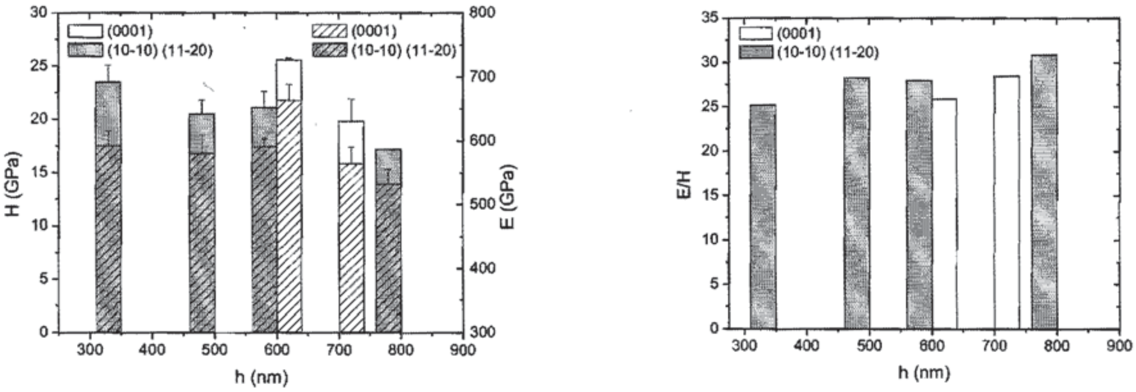


Figure 2. Cracks emanating from a cube corner indenter impression and the Palmqvist cracks generated.

Cracks were generated at differently oriented WC particles, by applying loads in the range of 5 to 25 g. After indentation tests, all the remnant imprints were inspected by means of SEM to measure the size of the contact impression and the lengths of the cracks emanating from its corners. The measured mechanical properties were correlated with the crystal anisotropy by assessing the crystal orientation of each tested WC grain by means of EBSD. The scan has been carried out with an acceleration voltage of 20 kV, a working distance of 12.5 mm, a detector distance of 174 mm, scanning a field corresponding to a magnification of X400 with a step size of 0.4 um. The EBSD detector, brand hkl, uses the "Chanel 5" software suit for data acquisition and processing, and is coupled to a field emission scanning electron microscope (FESEM), brand Zeiss, model Ultra Plus.

3. Results

The values of H and E obtained from measurements on WC single crystals are shown in Figure 3. Although a marked variation of the properties depending on the indented facet can be observed, these results are consistent with previous works [3,4]. Moreover, an indentation size effect (ISE) has also been observed. ISE may be described as an increase in hardness as the indentation size or load is reduced. Several reasons have been suggested to be responsible for ISE effect, particularly in terms of an increasing difficulty in inducing plasticity as the size of indentations is reduced or a rising contribution of elastic deformation as the hardness of the materials increases for small loads [8].



(a) (b)

Figure 3. Effect of the crystal anisotropy and ISE on: (a) H (plain bars) and E (pattern bars); (b) the ratio E/H in (0001) basal planes and (10-10), (11-20) prismatic facets.

The measure of K_C is experimentally difficult because either not all the indented grains show a well defined crack pattern or cracks propagate out of the indented grain. Thus K_C was calculated by selecting the indented grains with well developed cracks, in terms of size and shape. Table 2 shows the results of K_C obtained by using equation (1) for different WC crystals indented at different loads (Figure 4). Calculation of K_C following equation (1) depends on the ratio E/H; hence, the different mechanical behaviour of the different facets of WC should be taken into account when computing K_C. Ideally the values of E and H for each carbide should be taken to compute K_C. In this work, evaluation of H and E at the same penetration depth than the penetration depth required to generate fracture with the cube corner indenter was not possible. Main reasons behind it are the referred ISE and the fact that a cube corner tip produces a deeper imprint than a *Berkovich* tip. Thus, in the calculation of K_C (table 2) the ratio E/H is computed taking the value of E and H given in table 3 for each crystal facet.

Table 2. Fracture toughness of WC at different crystal orientation.

Indented plane	K_C ($MPa \cdot m^{1/2}$)
Prismatic facet	8.7 ± 1.1
Basal facet	7.2 ± 2.4

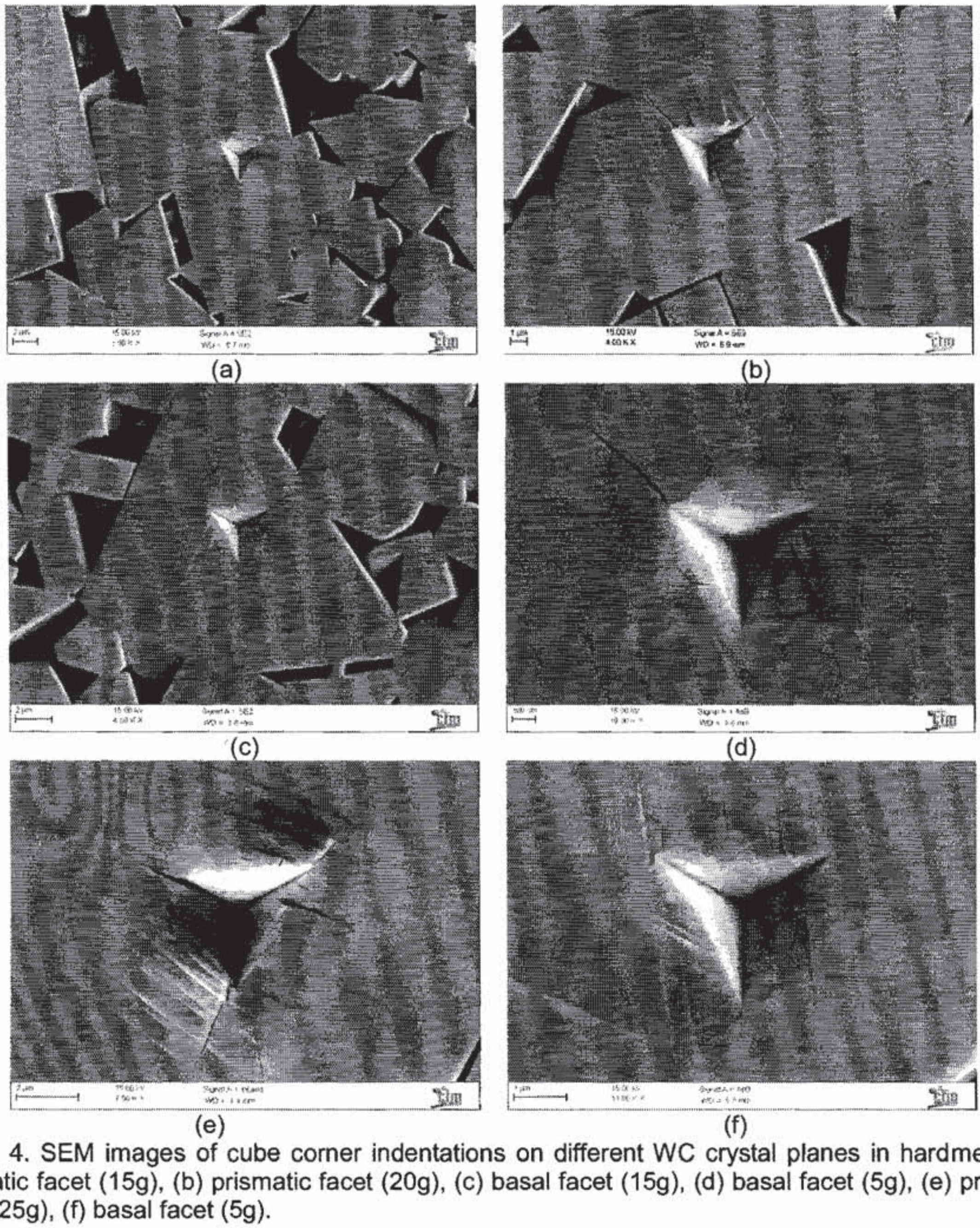


Figure 4. SEM images of cube corner indentations on different WC crystal planes in hardmetal: (a) prismatic facet (15g), (b) prismatic facet (20g), (c) basal facet (15g), (d) basal facet (5g), (e) prismatic facet (25g), (f) basal facet (5g).

Fracture toughness of metallic carbides has been evaluated by nanoindentation, giving values of about 2-5 $MPa \cdot m^{1/2}$ [6]. However, there are no available values of K_C for WC crystal embedded in a hardmetal. The values listed in table 2 for WC show relatively high toughness, 7.2-9.9 MPa·m$^{1/2}$, which are consistent with previous reported results, they are in the upper range of those either experimentally determined by Hertzian indentation [9] or linearly extrapolated, to zero volume fraction cobalt, from fracture toughness data for WC–Co alloys [10,11]. Such relatively high toughness measured by nanoindentation is also supported by the marked plastic deformation observed when indenting both prismatic and basal planes (Figure 4). Other metallic carbides present a brittle fracture, without evidences of plastic deformation [6]. This is a clear indication of the intrinsic tough-like character of the WC crystals, pointing out this as another of the microstructurally-related reasons for the success affiliated to WC-Co alloys as tool materials since their implementation almost one century ago.

When indenting the prismatic facets fracture is induced at both low and high applied loads (figures 4(a) and (b)). On the other hand, fracture emergence when indenting basal facets is dependent upon the applied load: in the low range (5g) cracks are generated (Figure 4 (d)), whereas in the high one (15 g) these features are suppressed (Figure 4(c)). Figures 4 (e) and (f) show a detail of the slip lines generated in a prismatic and a basal facet, respectively.

Table 3. Hardness anisotropy in WC crystals.

Crystallographic orientation	HV (Kg/mm^2) [4]	H (GPa)	E (GPa)
Prismatic facet	1450 ± 100	17.2 ± 0.1	564 ± 26
Basal facet	2500 ± 100	25.6 ± 0.2	532 ± 23

4. Discussion

Hardness in WC single crystals depends on the orientation of the indented crystallographic plane, as previously shown in other works [3,4,12]. In table 3 the results obtained by Pons et al. [4] with Vickers microhardness indentation at 20 g are shown along with the results obtained in this work at the same load. Moreover, French et al. also reported that hardness of basal facets is twice that determined on prismatic ones although hardness of basal facets shows little anisotropy and hardness of prismatic facets shows a marked orientation dependence [12]. Such marked anisotropy in hardness must be taken into account when analysing micromechanical properties of hardmetals.

Similarly to hardness, anisotropy is also observed in fracture toughness (table 2). It can be rationalized on the basis of the different plastic deformation capability ascribed to each crystallographic plane. Based in the slip traces around Knoop hardness indentations, French et al. [12] stated that in WC crystals the slip planes are {10-10} (prism planes) with <0001> and <1120> as preferred slip directions. Considering the effective resolved shear stress (ERSS) factor, which takes into account the most favourable slip systems adjacent to the indenter facets, it can be inferred that indentation of prismatic planes, as compared to the basal ones, should imply not only yielding at lower applied loads (and stresses) but also larger capability for developing plastic deformation before fracture [12-14]. This would finally result in a decrease of hardness and a rise in fracture toughness. The above ideas are consistent with the measured values of H and K_C when indenting prismatic and basal planes. Figure 4 shows slip traces around the indentation impression, always parallel to prismatic facets.

The particular single-, double- or multiple slip scenarios are given by the effective volume of material beneath the surface affected by the applied indentation load as well as the relative orientation of the corresponding slip systems in each case. Additionally, it could explain the different plasticity/fracture behaviour found in basal and prismatic planes at different indentation load. Taking into account that active slip planes are the prismatic planes, yielding in basal planes requires applied stresses even higher than those needed for inducing cracks as was shown in figures 4(c) and 4(d).

Finally, the above hardness/fracture toughness anisotropy together with the plasticity/fracture compromise should be considered for understanding the role of WC during crack propagation in hardmetals. In this regard, besides the relatively high toughness exhibited by this hard phase with independency of crystal orientation, the anisotropy factor may be speculated as another important microstructural factor for rationalizing the transgranular/intergranular interaction between propagating cracks and WC crystals. Research in this direction is currently in progress by the authors of this study [14].

5. Conclusions

Based on the results of micromechanical characterization by nanoindentation, together with the evaluation of crystallographic orientation by means of EBSD, the following conclusions can be drawn:

- The basal planes are harder than prismatic planes. On the order hand, in Young modulus has not been observed a strongly dependence on the indented facet.
- Fracture toughness of WC single crystal varies with the crystallographic orientation: indentation of prismatic planes gives higher toughness values than those obtained when indenting the basal ones. Prismatic facets have active slip planes that allow developing plastic deformation and thus increasing the fracture toughness measured when indenting prismatic planes.

This work shows that micromechanical characterization of WC, in terms of hardness and fracture toughness, together with microscopic analysis of the crystal orientation, allows the proper evaluation of the fracture resistance of WC taking into account the variability associated with crystal anisotropy. Such techniques can be seen as a powerful experimental tool to understand the crack-microstructure interactions on hardmetals, aimed at micromechanically designing tougher hardmetal grades.

6. References

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